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The organothallium phosphorus adduct $(Me_3SiCH_2)_3Tl{\bullet}P(SiMe_3)_3$ (**1**) was prepared by combining $(Me_3SiCH_2)_3Tl$ and $P(SiMe_3)_3$ at room temperature. Compound **1** was characterized by ^1H, $^{13}C\{^1H\}$, and $^{31}P\{^1H\}$ NMR, partial elemental analysis, EI mass spectrometry, and single-crystal X-ray analysis, the first to be reported for a thallium-group 15 adduct. Crystal data for **1**: trigonal system, space group P 3_1,, with a = 16.063(6) Å, c = 12.148(3) Å, D_{calcd} = 1.315 g cm^{-3}, and V = 2714.3(11) $Å^3$ for Z = 3. Refinement converged at R = 0.042 (R_w = 0.045). The Tl-P bond length in **1**, previously unreported for compounds of this type, was found to be 2.922(3) Å.

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Abstract

The organothallium phosphorus adduct $(Me_3SiCH_2)_3Tl{\bullet}P(SiMe_3)_3$ (**1**) was prepared by combining $(Me_3SiCH_2)_3Tl$ and $P(SiMe_3)_3$ at room temperature. Compound **1** was characterized by ^1H, $^{13}C\{^1H\}$, and $^{31}P\{^1H\}$ NMR, partial elemental analysis, EI mass spectrometry, and single-crystal X-ray analysis, the first to be reported for a thallium-group 15 adduct. Crystal data for **1**: trigonal system, space group $P\,3_1$, with a = 16.063(6) Å, c = 12.148(3) Å, D_{calcd} = 1.315 g cm^{-3}, and V = 2714.3(11) Å^3 for Z = 3. Refinement converged at R = 0.042 (R_w = 0.045). The Tl-P bond length in **1**, previously unreported for compounds of this type, was found to be 2.922(3) Å.

INTRODUCTION

As a result of our efforts to find facile methods of forming 13-15 bonds, we have prepared and characterized a variety of new and interesting compounds[1]. Among these are Lewis acid-base

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adducts of the type $R_3M\bullet E(SiMe_3)_3$ ($R = Me_3SiCH_2$, M = Ga, E = As or P;[1c] M = In, E = As[1a] or P[1c]; R = Ph[1a,b], M = Ga, E = As or P). The successful isolation of these compounds motivated us to continue our studies of 13-15 adducts, specifically those containing thallium. Although thallium-group 15 adducts have been prepared[2], no solid-state structures of such compounds have been reported.

To this end, herein, we report the synthesis and structural characterization of the novel organothallium phosphorus compound $(Me_3SiCH_2)_3Tl\bullet P(SiMe_3)_3$ (**1**).

EXPERIMENTAL

Synthesis

General Considerations. All manipulations of air- and moisture-sensitive materials were performed in a Vacuum Atmospheres HE-493 Dri-Lab containing an argon atmosphere or by general Schlenk techniques. Pentane and hexane were distilled over sodium-potassium alloy under dry dinitrogen. $(Me_3SiCH_2)_3Tl$[3], and $P(SiMe_3)_3$[4] were synthesized by literature procedures. ^1H, $^{13}C\{^1H\}$, and $^{31}P\{^1H\}$ NMR spectra were recorded on a Varian Unity 400 spectrometer operating at 400, 100.6, and 161.9 MHz, respectively. ^1H and $^{13}C\{^1H\}$ spectra were referenced to TMS by using the residual protons or carbons of deuterated benzene at δ 7.15 or 128 ppm, respectively. $^{31}P\{^1H\}$ spectra were referenced externally to 85% H_3PO_4 at δ 0.00 ppm. All NMR samples were prepared in 5-mm tubes which were septum-sealed under argon. Melting points (uncorrected) were obtained with a Thomas-Hoover Uni-melt apparatus and capillaries were flame-sealed under argon. Elemental Analyses were performed by E+R Microanalytical Laboratory, Inc., Corona, NY. Mass spectral data were collected on a JEOL JMS-SX 102A spectrometer operating in the electron ionization mode at 20 eV. X-ray crystallographic data were obtained at -120 ^oC on a Rigaku AFC6/S diffractometer utilizing graphite-monochromated Mo-Kα (λ = 0.71073 Å) radiation.

Preparation of $(Me_3SiCH_2)_3Tl{\bullet}P(SiMe_3)_3$ (1)

$(Me_3SiCH_2)_3Tl$ (0.466 g, 1.00 mmol) dissolved in 25 mL of pentane was added to a 250 mL round-bottomed screw-top flask equipped with a Teflon valve and magnetic stirbar. $P(SiMe_3)_3$ (0.251 g, 1.00 mmol) dissolved in 25 mL of pentane was added dropwise to the stirred $(Me_3SiCH_2)_3Tl$ solution. The resulting clear solution was stirred at room temperature outside of the dry box for 24 h. The volatiles were removed *in vacuo* to yield a faint-purple crystalline solid which was extracted into 5 mL of warm hexane. Cooling of the extract to -30 °C for 2 d afforded colorless X-ray quality crystals of **1** (0.562 g, 78.4%). mp. 68-71 °C. Anal. Calcd. (found) for $C_{21}H_{60}PSi_6Tl$: C, 35.20 (35.09); H, 8.44 (8.20). ^1H NMR (C_6D_6): δ 0.08, 0.90 [d, $-CH_2$, $^2J_{Tl\text{-}H}$ = 329 Hz], 0.13 [d, -SiMe$_3$, $^4J_{Tl\text{-}H}$ = 7 Hz], 0.33 [d, P-SiMe$_3$, $^3J_{P\text{-}H}$ = 5 Hz]. $^{13}C\{^1H\}$ NMR (C_6D_6): δ 2.07, 3.01 [d, $-CH_2Si(\underline{C}H_3)_3$, $^3J_{Tl\text{-}C}$ = 95 Hz], 4.13 [d, $-PSi(\underline{C}H_3)_3$, $^2J_{P\text{-}C}$ = 12 Hz], 29.02, 37.32 [d, $-\underline{C}H_2Si(CH_3)_3$, $^1J_{Tl\text{-}C}$ = 835 Hz]. $^{31}P\{^1H\}$ NMR (C_6D_6): δ –251.7 (s). Mass spec.: *m/z* 451, $(C_{11}H_{33}Si_3Tl)^+$; 379, $(C_8H_{22}Si_2Tl)^+$; 250, $(C_9H_{27}PSi)^+$.

X-Ray Crystal Structure Determination

Crystallographic data for **1** are summarized in Table 1. The crystal used was a colorless block which was mounted on a glass fiber with a viscous oil under a stream of cold dinitrogen. X-ray intensity data were recorded at -120 °C and the structures were solved by direct methods. Full-matrix least-squares refinement with weights based upon counter statistics was performed. Hydrogen atoms were incorporated at their calculated positions using a riding model in the later iterations of refinement which converged at R = 0.042 (R_w = 0.045). A final difference-Fourier synthesis revealed no unusual features (max. 1.64, min. -1.24 e Å^{-3}). Crystallographic calculations were performed using the NRCVAX[5] suite of structure determination programs. For all structure-factor calculations, neutral atom scattering factors and their anomalous dispersion corrections were taken from ref. 6. Interatomic distances and angles are given in Table 2. Non-hydrogen atom fractional atomic coordinates are listed in Table 3. An ORTEP[7]

diagram showing the solid-state conformation and atom numbering scheme of **1** is presented in the Figure.

RESULTS AND DISCUSSION

The 1:1 mole ratio reaction of $(Me_3SiCH_2)_3Tl$ with $P(SiMe_3)_3$ at room temperature affords the adduct $(Me_3SiCH_2)_3Tl{\bullet}P(SiMe_3)_3$ (**1**) in a good yield. Compound **1** is a crystalline material which is stable over long periods of time under inert atmosphere. In addition, **1** is surprisingly stable upon exposure to air and moisture and does not decompose rapidly. Interestingly, the resonances observed in the ^1H, $^{13}C\{^1H\}$, and $^{31}P\{^1H\}$ NMR spectra of **1** have chemical shifts that are very close to those observed for the starting materials, which suggests that **1** dissociates in benzene solution at room temperature. The ^1H and $^{13}C\{^1H\}$ spectra also evidence the fact that the thallium atom, with its spin of 1/2, couples to the protons and carbon atoms of its three Me_3SiCH_2- ligands (see references 2a and 2b for representative ^1H and ^{13}C spectral parameters of organothallium compounds).

Compound **1** crystallizes in the trigonal system with three molecules occupying the general positions of the $P3_1$ space group. In the solid state, this molecule adopts the conformation depicted in the Figure. The most noteworthy structural feature of **1** is the novel Tl-P bond length of 2.922(3) Å. This bond is longer than the sum of the covalent radii[8] (2.62 Å) for the thallium and phosphorus atoms, which is not surprising due to the dative nature of the bond. The coordination geometry about the thallium center is that of a distorted tetrahedron with a mean C-Tl-C angle of 118.0° and a mean C-Tl-P angle of 98.1°. Mean bond angles at the P atom [Tl-P-Si = 112.5° > Si-P-Si = 106.3°] reflect the relayed effect of the steric compressions resulting from the angular deformations around Tl. Since Tl-P analogs of **1** are not known, comparison of its bond lengths and bond angles with similar compounds is confined to the analogous gallium and indium compounds $(Me_3SiCH_2)_3Ga{\bullet}P(SiMe_3)_3$ (**2**)[1c] and $(Me_3SiCH_2)_3In{\bullet}P(SiMe_3)_3$ (**3**)[1c]. As expected, the metal-phosphorus bond lengths of 2.646(3) and 2.771(2) Å in **2** and **3**, respectively, are significantly shorter than the observed thallium-

phosphorus linkage in **1**. Surprisingly, the observed degree of angular distortion around the thallium center in **1** is in accord with that seen in **2** [mean C-Ga-C angle = 116.7°, mean C-Ga-P angle = 100.3°] and **3** [mean C-In-C angle = 116.2°, mean C-In-P angle = 101.4°] despite the metal size difference.

Future investigations in this area will focus on determining the utility of **1** as a single-source precursor to TlP and on preparing alternate organothallium pnicogen compounds.

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SUPPLEMENTARY DATA

Additional material consisting of a summary of crystallographic data and measurements, atomic coordinates, thermal parameters, bond distances and angles, and structure factors (9 pages).

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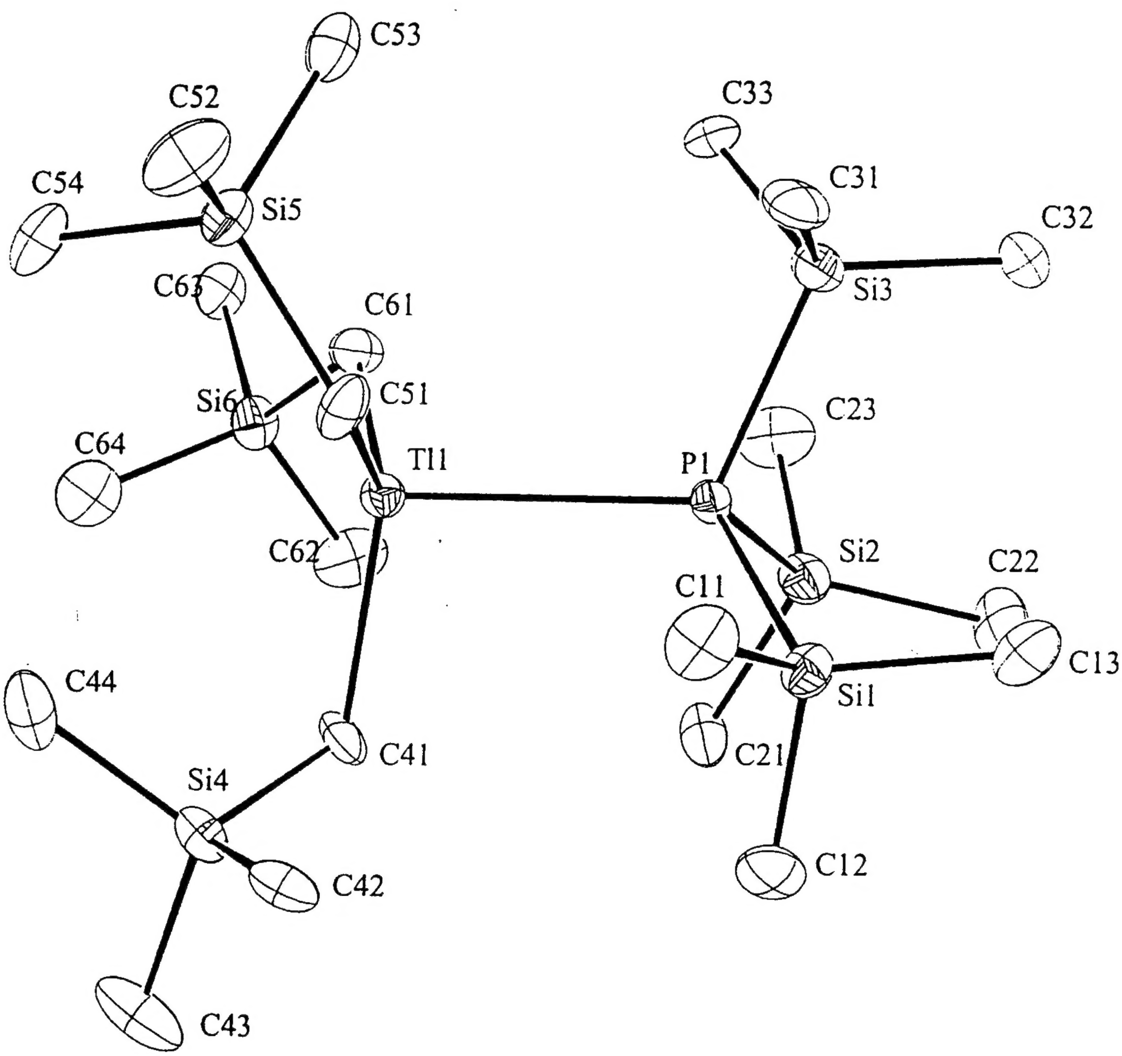
<u>Caption to Figure</u>

Figure. ORTEP diagram (30% probability ellipsoids) showing the solid state structure and atom numbering scheme of **1**. Hydrogen atoms are omitted for clarity.


C53
C52
C54
Si5
C33
C31
C32
Si3
C63
C61
C51
Si6
C64
Tl1
C23
P1
C62
Si2
C22
C11
C13
Si1
C44
C41
C21
Si4
C42
C12
C43

Figure

Table 1. Crystallographic Data and Measurements for $(Me_3SiCH_2)_3Tl{\bullet}P(SiMe_3)_3$ (**1**)

	1
molecular formula	$C_{21}H_{60}PSi_6Tl$
formula weight	716.55
crystal system	trigonal
space group	$P3_1$
a, Å	16.063(6)
c, Å	12.148(3)
V, $Å^3$	2714.3(11)
Z	3
radiation (wavelength, Å)	Mo-Kα (0.71073)
μ, mm^{-1}	4.75
temp, °C	-120
D_{calcd}, g cm^{-3}	1.315
crystal dimens., mm	0.30 x 0.30 x 0.30
T_{max}; T_{min}	0.360:0.300
scan type	ω
$2\Theta_{max}$, deg	50
no. of rflns recorded	3998
no. of non-equiv. rflns recorded	3998
no. of rflns retained, $I > 2.5\sigma(I)$	3333

Table 1 (continued)

	1
no. of params. refined	262
R; R_w[a]	0.042; 0.045
goodness-of-fit[b]	1.21
max shift / esd. in final least-squares cycle	0.012
final max, min $\Delta\rho$, e/Å^{-3}	1.64; -1.24

[a]$R = \Sigma(||F_o| - |F_c||)/\Sigma|F_o|$; $R_w = [\Sigma w (|F_o| - |F_c|)^2/\Sigma w |F_o|^2]^{1/2}$.

[b]Goodness-of-fit = $[\Sigma w\Delta^2/(N_{observations} - N_{parameters})]^{1/2}$.

Table 2. Bond Distances (Å) and bond angles (°) for **1**, with Estimated Standard Deviations in Parentheses

Bond Lengths

Tl(1)-P(1)	2.922(3)	Si(3)-C(32)	1.87(2)
Tl(1)-C(41)	2.24(1)	Si(3)-C(33)	1.84(2)
Tl(1)-C(51)	2.23(2)	Si(4)-C(41)	1.85(2)
Tl(1)-C(61)	2.25(2)	Si(4)-C(42)	1.85(2)
P(1)-Si(1)	2.267(5)	Si(4)-C(43)	1.87(2)
P(1)-Si(2)	2.249(5)	Si(4)-C(44)	1.85(2)
P(1)-Si(3)	2.261(5)	Si(5)-C(51)	1.86(2)
Si(1)-C(11)	1.85(2)	Si(5)-C(52)	1.88(2)
Si(1)-C(12)	1.86(2)	Si(5)-C(53)	1.87(2)
Si(1)-C(13)	1.84(2)	Si(5)-C(54)	1.86(2)
Si(2)-C(21)	1.84(2)	Si(6)-C(61)	1.82(1)
Si(2)-C(22)	1.86(2)	Si(6)-C(62)	1.87(2)
Si(2)-C(23)	1.84(2)	Si(6)-C(63)	1.86(2)
Si(3)-C(31)	1.87(2)	Si(6)-C(64)	1.85(2)

Bond Angles

P(1)-Tl(1)-C(41)	98.9(4)	P(1)-Si(3)-C(33)	107.7(5)
P(1)-Tl(1)-C(51)	95.1(4)	C(31)-Si(3)-C(32)	108.9(7)
P(1)-Tl(1)-C(61)	100.4(4)	C(31)-Si(3)-C(33)	109.4(7)
C(41)-Tl(1)-C(51)	116.5(5)	C(32)-Si(3)-C(33)	109.1(7)
C(41)-Tl(1)-C(61)	116.2(5)	C(41)-Si(4)-C(42)	109.3(7)
C(51)-Tl(1)-C(61)	121.1(5)	C(41)-Si(4)-C(43)	110.6(7)
Tl(1)-P(1)-Si(1)	110.7(2)	C(41)-Si(4)-C(44)	111.5(7)
Tl(1)-P(1)-Si(2)	113.7(2)	C(42)-Si(4)-C(43)	109.0(8)
Tl(1)-P(1)-Si(3)	113.1(2)	C(42)-Si(4)-C(44)	108.5(8)
Si(1)-P(1)-C(11)	105.8(2)	C(43)-Si(4)-C(44)	107.9(9)
Si(1)-P(1)-C(12)	107.1(2)	C(51)-Si(5)-C(52)	108.5(7)
Si(2)-P(1)-C(13)	105.9(2)	C(51)-Si(5)-C(53)	111.4(7)
P(1)-Si(1)-C(11)	109.9(5)	C(51)-Si(5)-C(54)	111.6(7)
P(1)-Si(1)-C(12)	108.7(5)	C(52)-Si(5)-C(53)	108.2(8)
P(1)-Si(1)-C(13)	114.2(5)	C(52)-Si(5)-C(54)	109.5(8)

Table 2, Continued.

C(11)-Si(1)-C(12)	109.5(8)	C(53)-Si(5)-C(54)	107.6(8)
C(11)-Si(1)-C(13)	107.3(8)	C(61)-Si(6)-C(62)	109.7(8)
C(12)-Si(1)-C(13)	107.1(8)	C(61)-Si(6)-C(63)	110.8(7)
P(1)-Si(2)-C(21)	108.1(5)	C(61)-Si(6)-C(64)	111.6(8)
P(1)-Si(2)-C(22)	112.9(6)	C(62)-Si(6)-C(63)	107.8(7)
P(1)-Si(2)-C(23)	107.9(5)	C(62)-Si(6)-C(64)	109.2(9)
C(21)-Si(2)-C(22)	109.5(8)	C(63)-Si(6)-C(64)	107.7(7)
C(21)-Si(2)-C(23)	109.0(8)	Tl(1)-C(41)-Si(4)	114.5(7)
C(22)-Si(2)-C(23)	109.3(9)	Tl(1)-C(51)-Si(5)	117.2(7)
P(1)-Si(3)-C(31)	108.1(5)	Tl(1)-C(61)-Si(6)	116.2(7)
P(1)-Si(3)-C(32)	113.5(5)		

Table 3. Non-Hydrogen Atom Fractional Coordinates and Equivalent Isotropic Thermal Parameters for **1**, with Estimated Standard Deviations in Parentheses

Atom	x	y	z	$B_{iso}(Å^2)^a$
Tl	0.33779(7)	0.01648(3)	0.7786(-)	2.21(4)
P(1)	0.32523(23)	-0.01183(24)	1.0169(3)	2.11(17)
Si(1)	0.1976(3)	-0.1566(3)	1.0616(3)	2.76(20)
Si(2)	0.4544(3)	-0.0102(3)	1.0908(4)	2.97(23)
Si(3)	0.3082(3)	0.1011(3)	1.1091(3)	2.74(20)
Si(4)	0.2590(3)	-0.1886(3)	0.6235(4)	3.16(23)
Si(5)	0.1911(3)	0.1084(3)	0.6955(4)	2.85(20)
Si(6)	0.5662(3)	0.1635(3)	0.6747(4)	3.15(21)
C11	0.0902(10)	-0.1757(11)	0.9849(14)	3.8(8)
C12	0.2257(11)	-0.2520(10)	1.0249(14)	3.9(8)
C13	0.1666(12)	-0.1708(11)	1.2089(14)	4.3(9)
C21	0.4859(12)	-0.0840(13)	1.0033(13)	3.9(11)
C22	0.4335(13)	-0.0568(14)	1.2342(14)	5.0(12)
C23	0.5543(11)	0.1144(12)	1.0896(17)	5.0(11)
C31	0.1836(11)	0.0777(11)	1.0855(13)	3.5(8)
C32	0.3295(12)	0.1019(11)	1.2609(13)	3.7(9)
C33	0.3950(12)	0.2191(10)	1.0513(13)	3.3(9)
C41	0.3473(11)	-0.1126(11)	0.7290(12)	3.3(8)
C42	0.1394(11)	-0.2599(10)	0.6882(13)	3.7(9)
C43	0.2946(13)	-0.2726(15)	0.5602(17)	5.9(12)
C44	0.2450(12)	-0.1152(13)	0.5116(13)	4.8(10)
C51	0.1944(10)	0.0059(10)	0.7622(13)	3.4(7)
C52	0.0621(11)	0.0773(12)	0.6823(16)	4.9(10)
C53	0.2557(12)	0.2201(12)	0.7797(14)	4.0(9)
C54	0.2481(12)	0.1349(12)	0.5567(14)	4.4(10)
C61	0.4744(11)	0.1588(10)	0.7663(13)	3.6(8)

$^aB_{iso}$ = the mean of the principal axes of the thermal ellipsoid

Table 3, Continued.

Atom	x	y	z	B_{iso}(Å^2)[a]
C62	0.6225(11)	0.0977(12)	0.7381(17)	5.1(11)
C63	0.6625(10)	0.2895(11)	0.6513(13)	3.7(8)
C64	0.5159(12)	0.1100(13)	0.5392(15)	4.8(10)

[a]B_{iso} = the mean of the principal axes of the thermal ellipsoid

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